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Anisotropic Liquids for Tunnel Detection

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WESTINGHOUSE ELECTRIC CORPORATION

TECHNICAL REPORT AFATL-TR-67-9 VOL 1

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ANISOTROPIC LIQUIDS FOR TUNNEL DETECTION VOLUME I

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FOREWORD

This report is Volume I of a two-volume final report prepared under Contract Number AF 08(635)-5262 by the Research Laboratories of the Westinghouse Electric Corporation, Pittsburgh, Pennsylvania. Work was performed on the contract during the period June 29, 1965 to July 29, 1966. Volume II of the final report presents the discussion of technical findings of the classified work program covered by Modification 2 of the subject contract and was prepared as a separate document. Volume I of the final report was prepared by L. C. Flowers, R. H. Calderwood and E. S. Bober. Volume II of the final report was prepared by K. W. Grossett, L. C. Flowers and E. S. Bober. All portions of the research work was done under the overall direction of Dr. Daniel Berg. This contract was initiated under ARPA Order 563.

The project was sponsored by the Advanced Research Projects Agency, Washington, D. C. and was conducted under technical direction of the Research and Technology Division, Air Force Armament Laboratory, Bio-Chemical Division (ATCC), Eglin Air Force Base, Florida. Lt. G. W. McCollum was the Project Engineer.

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JOHN E. HICKS, Colonel, USAF Chief, Bio-Chemical Division

ABSTRACT

The work reported herein is a sequal to previous studies under Contract Number AF 08(635)-4423 and reported in a Technical Report ATL-65-62 entitled "Thermal Detection of Underground Tunnels;" September, 1965.

In the present study, experiments were performed between August 12, 1965 and November h, 1965 to evaluate the feasibility of utilizing anisotropic liquid (AL) films to detect a tunnel one foot below ground and containing a heat source which simulated the thermal effects originating from a human being hidden in the tunnel. Soil temperature differences above the heated tunnel were most consistent in the early morning hours just before sunrise. The difference between the median soil temperatures at 4:00 AM above and outside the tunnel location ranged from 0.5°F in August to 3.0°F in November. The color responses of anisotropic liquid panels placed in windshield crossing the tunnel plot were generally in the proper direction, blues and greens indicating higher temperatures above the tunnel location and yellows, reds, and browns indicating lower temperatures outside the tunnel location. There were, however, serious difficulties with spurious color effects produced by temperature differences arising from natural causes. Mottled and variegated color patterns unrelated to the location of the tunnel always appeared when the anisotropic liquid panel was removed from the windshield and placed in direct contact with the soil. At the present time it seems doubtful that the naturally occurring variations in soil surface temperatures can be eliminated to the extent necessary for practical detection of a human being hidden in a tunnel.

Concurrent with the above study, a series of plastic films were evaluated for potential use in packaging the AL into a unit suitable for thermal mapping applications. The plastic films generally contributed to a lowering of the AL color appearance temperature with "Surlyn A," an ionomer film, exhibiting the least tendency. Several types of AL film packages were successfully fabricated using various techniques including heat sealing and vaccuum forming.

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SECTION I

INTRODUCTION

Tunnel detection by means of anisotropic liquid films was previously studied under Contract No. AF 08(635)-4423. The results of this study were described in a Technical Report ATL-TR-65-62 "Thermal Detection of Underground Tunnels", September 1965.

The most significant conclusion reached in this previous work was that tunnel detection based on the color change of an anisotropic liquid film was possible when the tunnel contained a heat source sufficient to establish a definite pattern of temperature difference on the surface of the overlying soil. In the absence of an internal heat source, the naturally occurring soil temperature pattern was apparently unrelated to the presence of the tunnel cavity below ground, and in that case, the location of the tunnel was not detected by soil surface explorations with anisotropic liquid films.

The work program that was pursued under Contract No. AF 08(635)-5262 is a continuation of the feasibility study program initiated under the previously mentioned Contract No. AF 08(635)-4423 to evaluate the feasibility of utilizing anisotropic liquids, or "liquid crystals" as these liquids are sometimes called, for detecting temperature differences. One objective in the program continuation thus far has been to investigate the application of anisotropic liquid films particularly in respect to the detection of thermal effects originating in tunnels or other underground cavities. Since this limited objective has now been reached, that portion of Contract No. AF 08(635)-5262 dealing with tunnel detection may be considered essentially complete as described in this final report.

The tunnel detection study program consisted of two parts:
(1) determination of limiting conditions under which heat sources in the tunnel could be detected, and (2) investigation of packaging materials suitable for anisotropic liquid film applications in field use. These parts are reported separately in the text which follows.

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SECTION II

DETECTING THERMAL EFFECTS ORIGINATING IN TUNNELS

One possibility suggested by our previous experiments (1) was that human beings hidden in tunnels or underground cavities would constitute sources of heat which might be detectable by soil surface exploration with anisotropic films. An average male adult seated at rest liberates 114 watts; if seated but moderately active, he liberates 139 watts. During a slow walk his heat output is 161 watts and at a faster walk, 3 mph, it reaches 293 watts. Intermittent heavy work as with a pick and shovel causes him to give off heat at a rate between 440 and 586 watts.

The previous experiments with anisotropic liquid films had successfully detected the presence of a simulated underground tunnel and its precise boundaries when a 600-watt source of electrical heat was placed in the tunnel and the depth of soil cover over the tunnel was 8 to 9 inches. The simulated tunnel in this instance consisted of a buried length of hollow, unglazed clay pipe six feet long and two feet internal diameter. The wall of the pipe was approximately two inches thick and its topmost point was 8 to 9 inches below ground level. The air inside the tunnel had reached a temperature of 123°F as a result of the 600watt heating and was at that temperature at the time the soil temperature detection experiments were made. The open air temperature above ground was 54°F during these experiments. The small size of the simulated tunnel, its shallow depth below ground, and the relatively hot air produced by the electric heater were, of course, conditions more favorable for detection than would be expected in the case of human beings hidden in actual tunnels. A new experimental arrangement which provided a more realistic situation was therefore constructed.

2.1 EXPERIMENTAL CONDITIONS AND INSTRUMENTATION

2.1.1 Tunnel Construction

The so-called "new" or "second" tunnel described previously (1) was again put to use in the present work. To review briefly, the tunnel was driven during the last part of October and early November, 1964, into sloping ground which was then levelled off to a uniform overlay of undisturbed soil with its top surface 12 inches above the inside ceiling of the tunnel. The tunnel was framed and timbered to prevent "cave-in", and the resulting underground cavity was approximately the shape of a rectangular parallelopiped 30 inches wide by 51 inches high and 7 feet long. It was necessary to clean out debris and repair general damage caused by winter and spring weather before the tunnel could be reused;

however, care was taken in restoration to keep the dimensions of the tunnel and the plot of soil above the tunnel the same as before. The enclosed space in this tunnel is approximately four times as large as the space in the simulated tunnel used in the previous 600-watt heater experiments. A diagram of the tunnel construction and heating arrangement, and the positions for temperature detection or measurement on the soil surface above the tunnel are shown in Figure 1.

2.1.2 Source of Heat in Tunnel

The source of heat inside the tunnel was designed to simulate the body temperature as well as the heat output of human beings. For this purpose, a 20-gallon galvanized tank containing 112 pounds of water was placed inside the tunnel at the position indicated in Figure 1. Electric heat was supplied to the water tank by a 20-ohm immersion heater (Fisher Scientific Co. Cat. No. 11-463-5) having 50 inches of active heating length and controlled by a calibrated Variac located outside the tunnel. A small motor-driven stirrer was mounted near the immersion heater to circulate the water and maintain a uniform temperature. A 16-inch table fan installed inside the tunnel about 2 feet away from the tank and blowing directly on the tank provided air convection currents which balanced the heat input so that the water temperature stabilized reasonably close to the temperature of the human body. The tunnel mouth was closed with a plywood cover which was not removed or disturbed until both surveys had been completed.

A preliminary test with the foregoing arrangement in August indicated that the water temperature would stabilize fairly close to 99°F when the immersion heater was energized continuously at 350 watts as measured by a wattmeter. The Variac in the heater line was used to set the currentvoltage relation in the preliminary test and the same setting was used in subsequent experiments as long as the weather remained warm. The fluctuation in the water temperature over a 24-hour period was generally from 98°F to 102°F. These temperature limits were fairly well maintained during the course of the experiments which ran from August into November; however, on September 30 it was necessary to compensate for generally lower outdoor temperatures by increasing the immersion heat input to 420 watts. The fan and stirrer motor added approximately another 50 watts to the heat entering the tunnel, hence the total heat input was about 400 watts in the early tests and about 470 watts in the later tests. As has been noted, such wattages should simulate the heat output of a human being under conditions of rather strenuous activity.

The experiments in August were performed in one case with the heat source "on" and in the other case, with the heat source "off". During September and October the heat source was allowed to operate continuously but no further change in the control setting was made after the increase to 470 watts on September 30. On the day of the final test, November 4, the water tank temperature ranged from 93° to 95°F which, for the purpose of the experiment, was still regarded as a reasonable simulation of human body temperature.

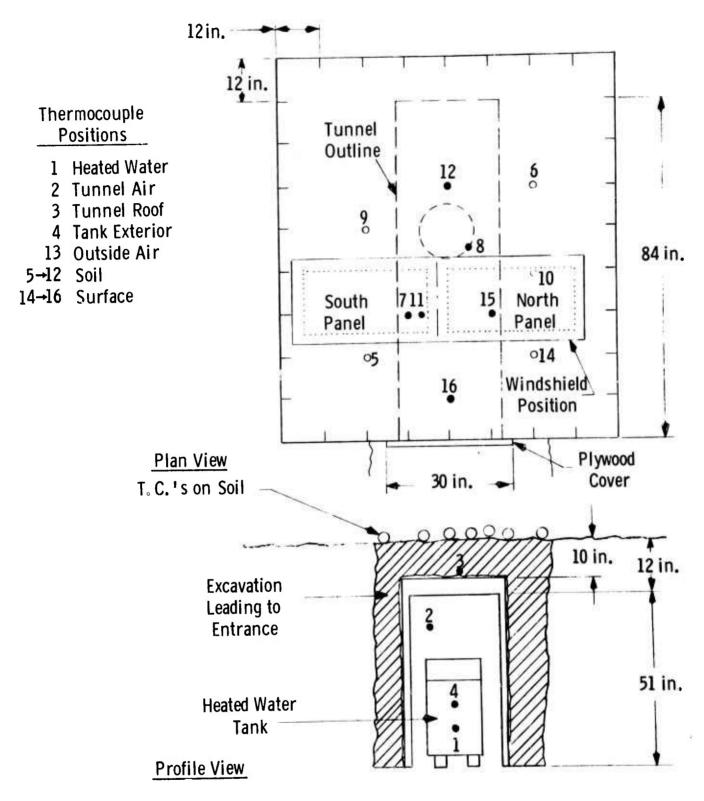


Fig. 1—Tunnel plot showing position of heating and testing equipment

2.1.3 Temperature Surveys

Temperatures were measured by 16 thermocouples connected to a Speedomax recording potentiometer. Reference junctions were immersed in melting ice. The recorder was adjusted to print out at 5 minute intervals. The maximum deviation recorded when all 16 thermocouples were immersed in water at 76°F was -0.03 mv, which corresponds to approximately 1°F. To reduce this error, the deviations from the mean were applied as correction factors to each separate thermocouple reading in posting the data and plotting graphs.

The positions of the thermocouples in respect to the location of the tunnel are shown in Figure 1. Eleven thermocouples were placed on the soil surface, five being distributed outside the tunnel outline and six inside the tunnel outline. These thermocouples were stapled to the soil surface in such a manner that the beads were in firm contact with the ground but with their upper surfaces exposed to ambient air. The beads were encapsulated in insulating (Picein) cement to guard against electrochemical effects.

The five remaining thermocouples were located as follows: T.C. #1 immersed in the continuously stirred water tank at a fixed position; T.C. #2 suspended in the tunnel air at a point beyond the direct blast of air from the fan; T.C. #3 inserted from inside the tunnel through the planks which formed its "roof" and making contact with the bottom surface of the overlying soil; T.C. #4 attached to the outside wall of the tank at a point facing the fan; and T.C. #13 suspended in the open air about 3 feet above ground and off one side of the tunnel location.

The Speedomax recorder was operated continuously for at least 24 hours in each experiment. Two exploratory temperature surveys were performed in August, one with the heat source operating in the tunnel and the other with the heat source turned off and cooling to ambient temperature. Three more surveys were made in October and November, in each case with heat on" in the tunnel. The purpose of these later surveys, however, was mainly to assist in the interpretation of the color response of anisotropic liquid panels which were then being studied at various positions on the tunnel plot.

2.1.4 Development of Anisotropic Liquid Mixtures

A series of ten anisotropic liquid mixtures with overlapping color-temperature transition ranges was developed for the tunnel detection studies. The overall temperature range covered by these ten mixtures was from about 30°F to about 70°F and each individual mixture was designed to pass through its characteristic color spectrum within a temperature bracket of 5° to 7°F. The mixture compositions and nominal color transition ranges are given in Table I.

Table I

Composition of Anisotropic Liquid Mixtures Designed for Tunnel Detection Experiments

	(OF)	(28°-41°)	(37°-43°)	(36°-46°)	(46°-52°)	(50°-57°)	(55°-61°)	(26°-64°)	(62°-68°)	(66°-70°)	(68°-73°)
អ្ន	00	-2° to +5°	3° to 6°	4° to 8°	8° to 11°	10° to 14°	13° to 16°	15° to 18°	17° to 20°	19° to 21°	20° to 23°
G.P.	(Note 5)	1	1	1	1	11,0	0.6	0.6	0.6	0.6	0.6
Percent C.N.	(Note 4)	1	1	;	!	15.0	16.0	19.0	22.0	25.0	28.0
ion - Weight	(Note 3)	20.0	1	1	1	l	1	:	1	;	1
Composit	(Note 2)	1	20.0	17.5	15.0	Ī	1	1	1	;	:
Code 0.C.C. C.G.C. C.C.C. C.N.	(Note 1)	80.0	80.0	82.5	85.0	0.47	75.0	72.0	0.69	0*99	63.0
Code	Number	2841	3743	3946	7655	5057	5561	5964	6268	0299	6873

0.C.C. = Oleyl cholesteryl carbonate having a green transition at 22. $^{4}{}^{\circ}\mathrm{C}$. (Prepared at Westinghouse Research Labs). Note 1.

Note 2. C.G.C. = Cholesteryl geranyl carbonate (Eastman No. 921371).

Note 3. C.C.C. = Cholesteryl crotyl carbonate (Eastman No. 921362).

C.N. = Cholesteryl nonanoate (Prepared at Westinghouse Research Labs.) Note 4. C.P. = 'Cholestery' propionate (Prepared at Westinghouse Research Labs.) Note 5.

The visual color responses of the anisotropic liquid mixtures were determined at 1°C temperature intervals with the apparatus shown in Figure 2. In using this apparatus, one watches the anisotropic liquid (AL) film sample while it is being slowly cooled (or heated) under close temperature control and records the colors which appear at the desired temperature intervals measured on the Type K-3 potentiometer. A transparent polyethylene bag containing dry nitrogen (N₂) gas at a slight positive pressure is used to prevent condensation of atmospheric moisture on those samples which must be cooled below the dew point for their characteristic colors to appear. The AL film colors are judged and recorded according to their closest match to the set of color standards in the ISCC-NBS Centroid Color Chart, National Bureau of Standards' Standard Sample No. 2016. All observations to date have been made in ordinary room light (partly daylight and partly fluorescent light).

The AL film samples used in the color response determination were prepared in the usual manner by casting from solution onto a blackened "Mylar" base. The ingredients of the AL mixture to be tested were first weighed out in the desired proportions according to Table I and in sufficient total amount to make a 10 weight per cent solution in 25 grams of solvent mixture. The solvent mixture consisted of 3 parts by volume petroleum ether (boiling range 36.7°C - 53.4°C) and one part by volume chloroform. A small amount of each solution was used in the color response determination and the major portion was saved for possible later use in preparing anisotropic liquid panels for the tunnel detection experiments.

As indicated in Figure 2, the AL film sample is supported by a "Mylar" base. The 'Mylar" base is about 0.00025 inch thick and is supported on a relatively light-weight brass hoop having an inside diameter of 3.75 inches. The bottom surface of this 'Mylar" base is sprayed with black acrylic lacquer "Krylon" before the AL film is applied to the top surface. The black background eliminates spurious light effects and allows only the reflected scattering colors to be seen. A pipette or medicine dropper is used to introduce approximately 0.75 ml of the AL solution at the center of the hoop; this volume is sufficient to flow over the entire area (71 cm²) of the 'Mylar' base inside the hoop. The solvent is allowed to evaporate for about five minutes at room temperature and the film is then rapidly heated through its clearing point by a 250-watt reflector-type heat lamp shining from above. This rapid heating completes the removal of the solvent and smooths out minor imperfections.

The results of the color response temperature determinations are shown in Table II. In all of these measurements, the test was begun at a temperature above the clearing point of the AL film and the sample was cooled through its color transition phase. When the sample began to show a color response, the practice was to allow the temperature to dwell (i.e., oscillate) for two to five minutes at each degree point before cooling the sample through the next 1°C interval. This allowed sufficient time for a fairly reliable color match to be established.

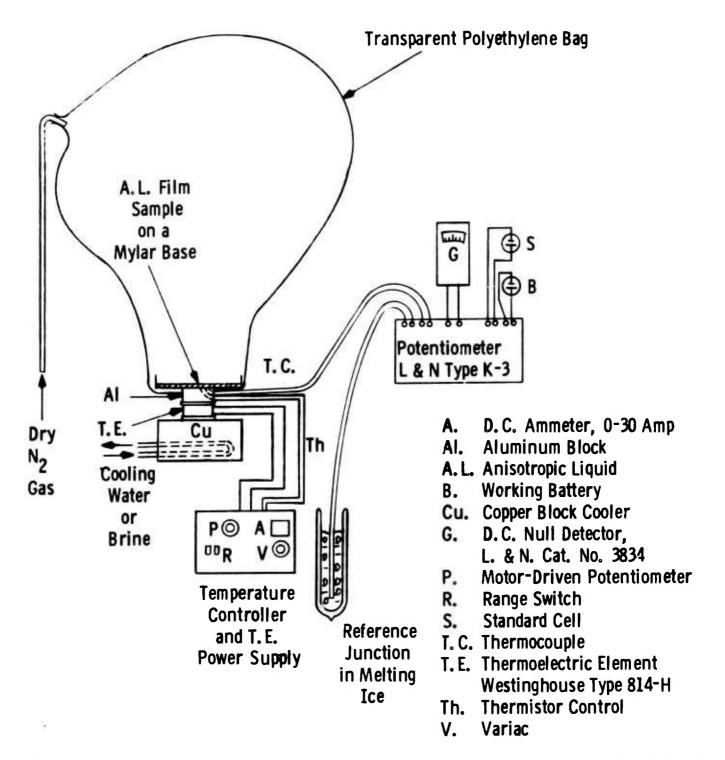


Fig. 2—Apparatus for determining visual color response temperatures of anisotropic liquids

Table II. Color Response of Anisotropic Liquid Mixtures Developed for Tunnel Detection Studies

		2020	12904	1,500, 10000, 40000	1727	7.00	13740	13/45		
ζ	00101000	colorless	colorless	colorless	colorless	colorless	colorless	colorless	colorless	
,		=	=	:	=	£		£	£	
		*	=	=	=	=	=	=	=	
0	deep B.	:			:	=	=	=	£	
	deep G.	vivid pB.	=	=	:	: :	ı,	=	=	
-	v. deep R.	s. B .	F	=	=	=	:	:		
	Black	deep YG.	vivid pB.	E	= 1	=	=	:	: :	
	E	d. r Br.	deep G.	deep B.	:	=	= }	=	: :	
	=	Black	deep R.	wivid pB.	deep B.	=	=	£	=	
	=	=	d. r Br.	s. B.	vivid pB.	=	•	=		
	:	=	Jack	deep YG.	s. B.	£	=	z	=	
	£		=	vivid G.	deep YG.	=		=	÷	
	=		ŧ	Black	vivid G.	d. pB.	=	=	=	
	-	:	:	ŧ	v. deep R.	deep pB.	E	:	÷	
		:	=	=	Black	vivid G.	=	=	z	
	: :	:	=			deep r. Bl.	E	=	=	
	: :	:	:	:	ŧ	. E	d. pB.	=	=	
	:	:	: :			10010	return DR	d. nB.	:	
	=	£	.	:	: :	Black	ed pivis		a c	
	:	=	=	=	=		vivid G.	ATATO DE		
	=	=	c	=	E	=	d. r Br.	vivid G.	deep pB.	
	E	ŧ	=	r	=	E	r. Bl.	s. r Br.	vivid pB.	
	=	=	=	:	=	£	Black	d. r Br.	d. gB.	
	:	=	E	E	£	=	:	r B1.	vivid G.	
	=	=	:	ŧ	:	=	=	Black	vivid yG.	
		i	=	=	E	=	=	=	s. r Br	
	÷		l		=	E	=	r	d. r Br	
	=	=	:	:	; ;	: :	=	=	, i	
	=	E	=	=	= 1	: :	: :	: =	d. r br	
	F		=	=	E	=	=	:	61 K.	

List of Abbreviations and Color Numbers According to ISCS-NBS Color Name Charts

v. deep R. = very deep red, No. 1 ⁴ bl R. = blackish red, No. 21 d. rBr = dark reddish brown, No. 4 ⁴ s. rBr = strong reddish brown, No. 4 ⁰ r. Bl. = reddish black, No.2 ⁴	
deep G. = deep green, No. 182 vivid G. = vivid green, No. 139 deep YG. = deep yellow green, No. 118 d. yG. = dark yellowish green, No. 137 vivid yG = vivid yellowish green, No. 129 deep R. = deep red, No. 16	
deep B. = deep blue, No. 179 s. B. = strong blue, No. 178 deep pB. = deep purplish blue, No. 197 d. pB. = dark purplish blue, No. 201 vivid pB. = vivid purplish blue, No. 194 d. RB. = deep purplish blue, No. 194 d. RB. = deep purplish blue, No. 197	

2.1.5 Anisotropic Liquid Panels

Two rectangular panels similar to those described previously (1) were again used in the present study. As before, the panels were coated lengthwise with parallel strips of three or four different anisotropic liquid mixtures. The mixtures were selected so as to have overlapping color response temperatures according to Table II and thus each panel could be used over a total range of about 10° to 12°F variation in soil temperatures. The panel base was a 0.00025 inch "Mylar" film stretched over a 21 inch x 37 inch aluminum window screen frame from which the screening had been removed. Barriers of masking tape were used to divide the 21 inch width into longitudinal stripes. The "Mylar" bases were painted black on their bottom surfaces before the anisotropic liquids were cast onto the top surfaces. Covers cut from clear Plexiglas sheet 0.125 inch thick were bolted to the aluminum frames to prevent the anisotropic liquid film from becoming contaminated by air-borne dust and also to make a rigid structure less likely to be damaged in handling. 'The 'Mylar" base was further supported at its mid point by a horizontal Plexiglas strut 0.5 inch wide and 0.125 inch thick inserted between the base and the cover. A panel so constructed is shown in the photographs color contour maps to be discussed later.

2.1.6 Use of Windshield to Protect Panels

It has been our general practice to employ two methods of panel placement on the soil surface of the tunnel plot. The simplest method, of course, is to place the panels in direct contact with the soil and observe the colors that appear at different positions in relation to the location of the tunnel. This method, however, was never really satisfactory with the panels as constructed. The color response was always erratic when the panels were laid directly on the ground and irregular patches of intermingled colors would appear instead of a definite pattern as desired. There seemed to be at least four reasons for this. First, the soil surface was not uniformly flat and the small irregularities in contour tended to push the thin "Mylar" film which constitutes the bottom of the panel upward so that the anisotropic liquid film touched and smeared over the Plexiglas cover plate. This always produced a spurious color effect. Second, the aluminum frame of the panel touched the ground at some points and was off the ground at other points. This could result in uneven thermal gradients in the frame which in turn could cause uneven variations in the temperature of the adjacent anisotropic liquid film. Third, the bottom of the panel also made irregular contact with objects on the soil surface, revealing the shape of small stones, leaves, moist areas, etc. which produced a mottled color effect in most instances. Finally, the panel placed directly on the ground is exposed to air currents which may have a greater effect on its temperature than does the underlying soil.

The preferred method insofar as reliability of observation is concerned is to place the panels in a "windshield" that extends across the tunnel boundaries on the test plot. This method is shown on Figure 1. The windshield minimizes changes in temperature due to air currents and also supports the panel slightly above the soil surface so that many of the difficulties encountered in direct contact are avoided. (1) and is again illustrated in Figure 3 of the present report.

2.2 RESULTS AND DISCUSSION

Four separate experiments were performed at the tunnel plot during the three-month testing period. The results of these experiments will now be presented and discussed in chronological order.

2.2.1 First Soil Temperature Surveys; August 11-12, 1965

The heating arrangement described in Section 2.1.2 was installed in the tunnel and put into operation about 48 hours before the test was scheduled. This "preheating" allowed the heat input and output to reach fairly "steady state" conditions by the time the temperature surveys were started. After recording temperatures for 24 hours under these conditions, the immersion heater was disconnected and recording was continued for an additional 24 hours while the water tank cooled to ambient temperature. The fan and stirrer were kept running, hence there was still about 50 watts heat output in the tunnel during this cooling period.

The tunnel plot arrangement conformed generally to Figure 1 except for the windshield which was not yet in place and the thermocouple positions which differed slightly as shown in Figure 4. In these first surveys, the soil surface thermocouples were equally divided, six inside the tunnel outline and six outside the tunnel outline and no thermocouple was placed on the exterior of the water tank. The data recorded in the "heat-on" and "heat-off" periods are represented in Figures 5 and 6, respectively. To conserve space and avoid confusion in the graphs, the data are plotted only at 20-minute intervals although the recorder provided separate read-outs at 5-minute intervals. Even so, the graphs became very confused when an attempt was made to plot the 12 soil temperatures individually at a 20-minute spacing. The temperatures inside and outside the tunnel outline did not separate neatly into two groups, one with higher temperatures than the other, as had been hoped for. There was always considerable intermingling of the individually plotted temperatures because of the scatter within each group, and when the scatter diminished, as it did in the early morning hours, the 12 soil temperatures clustered together within a 3° or 4°F range which was apparently the same for points outside the tunnel outline as for points inside the tunnel outline. It was possible, however, to show a difference between the two groups by plotting the median temperatures in each set of six as has been done in Figures 5 and 6.

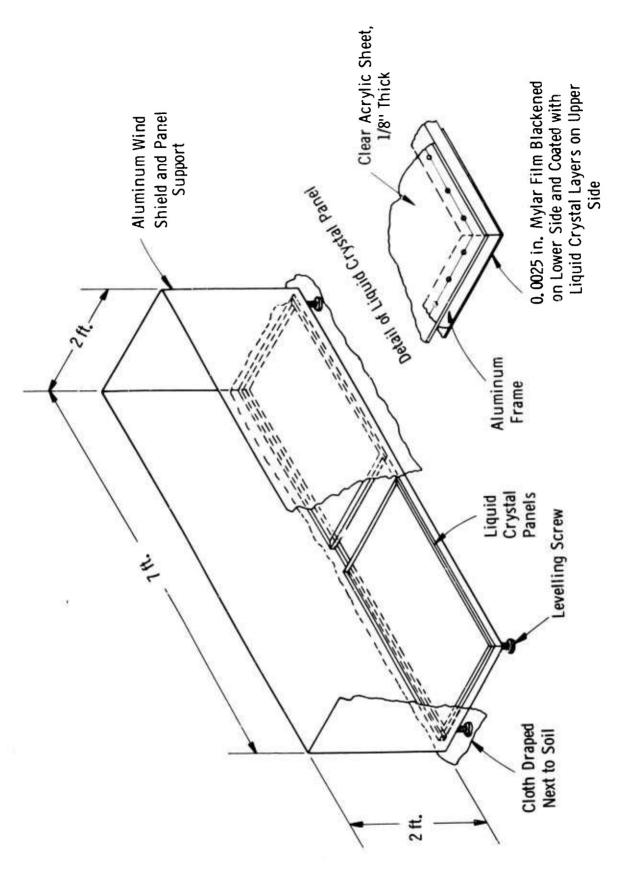


Fig. 3—Anisotropic liquid panel arrangement for tunnel detection experiments

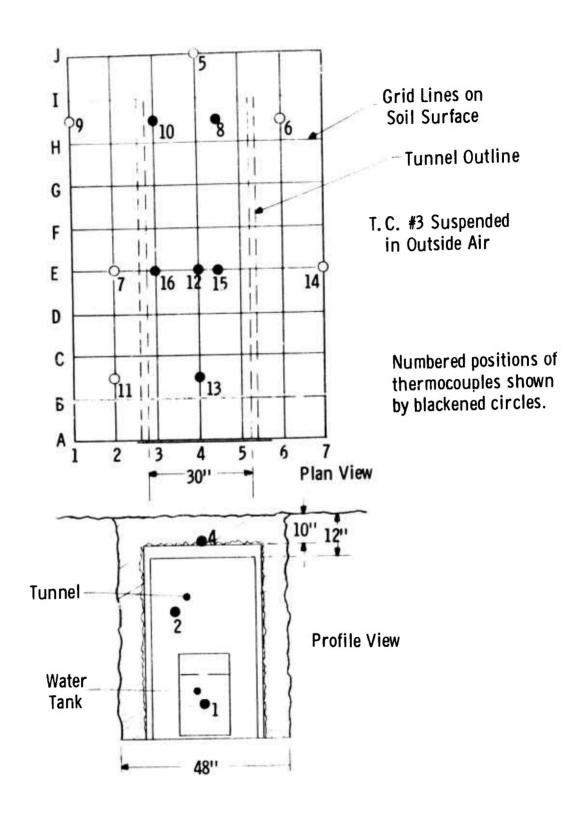


Fig. 4-Tunnel and location of thermocouples in first two temperature surveys

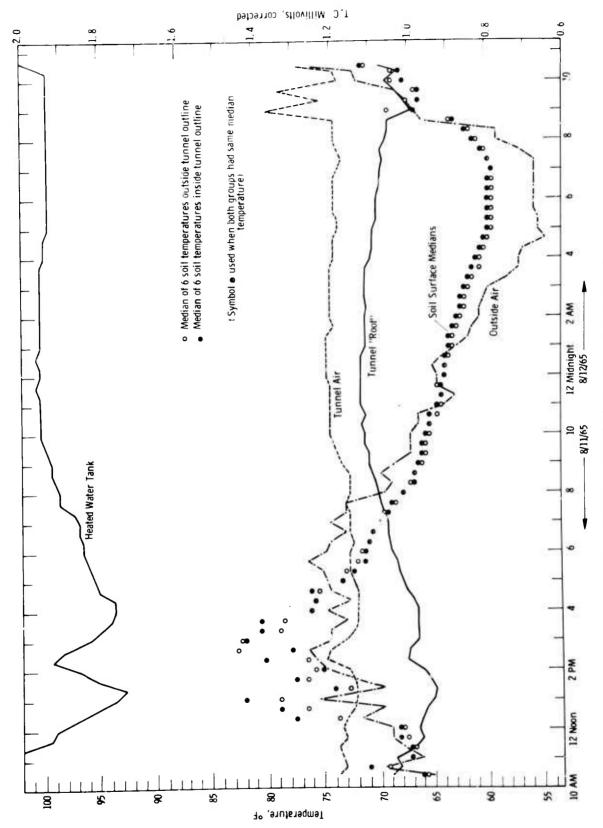
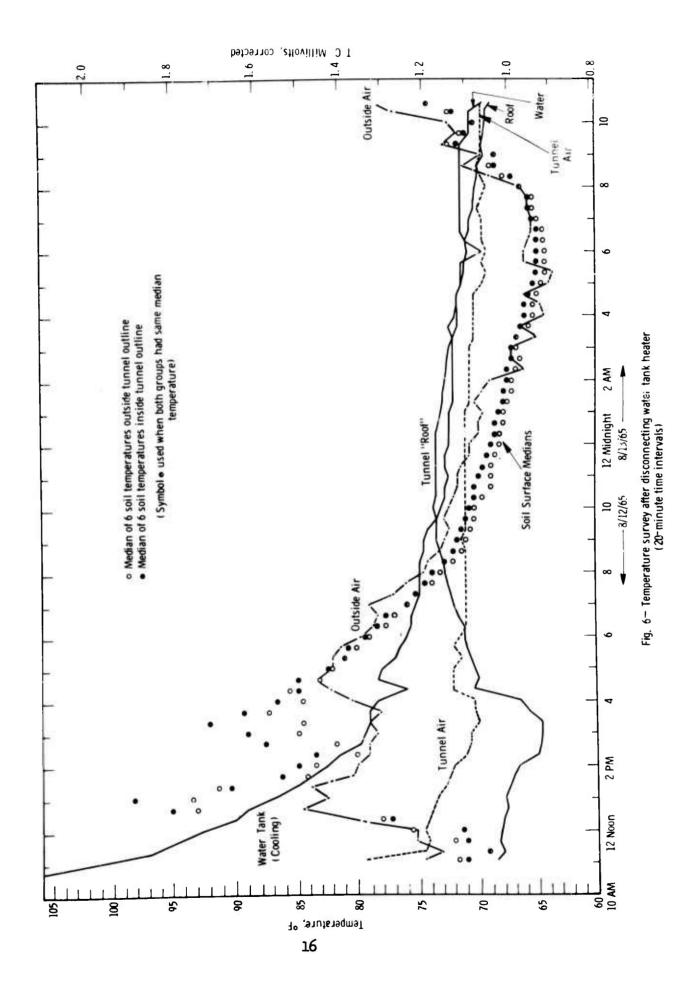


Fig 5—Temperature survey with 400 watts continuous heat in tunnel (20-minute time intervals)



The graph for the "heat on" period shown as Figure 5 indicates that human body temperature was reasonably well simulated by the water temperature. The lowest recorded water temperature was 92.5°F and the highest, 109°F. Both of these extreme values occurred within the first three hours after recording was started and can be attributed at least in part to disturbances created by opening the tunnel and entering it to install the thermocouples at the start of the test. Conditions inside the tunnel seemed to stabilize after seven or eight hours and from 8:00 P.M. until 10:00 A.M. the next morning the water temperature remained quite steady between 99°F and 101°F.

The temperature of the tunnel air also remained quite steady at about 74°F for most of the "heat on" period. During the first 23 hours of this 24-hour survey, the total variation was only a little over 2°F. The tunnel was opened and entered shortly after 8:00 A.M. to make adjustments for the "heat off" period to follow. This disturbance apparently caused the tunnel air temperature to rise and then fluctuate in approximately 5°F cycles. However, this fluctuation coming near the end of the "heat on" survey does not interfere with interpretation of the significant portions of the data.

The temperature of the air inside the tunnel was thus uniformly about 25°F lower than the water tank temperature during the greater part of the survey. One can thus infer from this "steady state" condition that heat was leaving the tunnel at the same rate at which it was being put in electrically, i.e., 400 watts. This heat would be expected to escape in all directions, part through the tunnel floor, part through the tunnel walls and part through the tunnel ceiling or "roof". In the absence of external work, heat can flow only from a hotter to a cooler body. Thus if heat is leaving the tunnel in a direction through the "roof" and into the overlying layer of soil, the upper surface of the "roof"—i.e., the point of contact between the soil cover and the roof planks — should be at a lower temperature than the tunnel air. Figure 5 shows that this is indeed the case. During the time of day of most interest to us, namely, the hours between 8:00 P.M. and 8:00 A.M., the "roof" was 2°F to 4°F cooler than the tunnel air.

There is also a curious diurnal swing of the "roof" temperature which appears to be more variable than the tunnel air temperature. The "roof" temperature seems to reach its lowest point during the middle of the day --1:00 P.M. to 3:00 P.M. -- when the outside air is hottest and its highest point around midnight when the outside air has become much cooler. This effect can be observed in both graphs regardless of "heat on" or "heat off" conditions. The tunnel "roof" temperature is, of course, also the ground temperature 10 inches below the soil surface. At this depth of soil, as previously shown by Brooks et al the temperature generally follows the diurnal swings of the natural air temperature above the soil, but with much less amplitude and roughly 8 to 12 hours out of phase.

It can be seen in Figure 5 that heat is flowing upward out of the tunnel through its "roof" at all times during the "heat on" survey.

However, there is only a certain portion of the day during which this heat can continue its upward flow to reach the soil surface 10 inches above the "roof". That is during the night and early morning from about 8:00 P.M. to 8:00 A.M., when the soil temperatures were lower than the "roof" temperatures. During the other half of the day, particularly from about 10:00 or 11:00 A.M. to 7:00 or 8:00 P.M., soil surface temperatures were generally higher than the "roof" temperature, in which case heat would be flowing downward from the soil surface toward the tunnel "roof".

Thus during these daylight hours heat was flowing into the layer of soil above the tunnel from two opposite directions: - one, from the tunnel air upward through the tunnel "roof" and the other, from the soil surface downward through the soil layer. A minimum temperature should therefore exist at some intermediate point within the soil layer. This point of minimum temperature presumably lay fairly close to the soil surface when the soil temperature first began to exceed the "roof" temperature, but moved slowly downward during the day to some unknown depth, then reversed its direction and moved slowly upward until shortly after 7:00 P.M. the soil temperatures were again below the tunnel "roof" temperature.

It can be assumed that a natural point of minimum temperature also moved up and down below the soil surface outside the tunnel outline. Otherwise the out-of-phase diurnal oscillation reported by Brooks would be difficult to explain. It was therefore reasoned that the marginal condition for detecting a tunnel by soil surface temperature exploration would be determined by the effect of the contained heat source on the magnitude, a location, or both magnitude and location of the natural minimum temperature point. The small differences between the median soil temperatures plotted in Figures 5 and 6 indicate that the conditions employed in these experiments may indeed be marginal.

The most pronounced temperature differences seems to have occurred during daylight hours, i.e., when the tunnel "roof" is coolest and it can probably be assumed that the outside soil at the same depth would have been at an even lower temperature. Unfortunately, this is just the time when the soil temperatures show extreme variations due to sunlight and other natural influences. On the other hand, the most consistent median temperature differences, though usually less than 1°F, occurred during the night and early morning. In this respect there seems to be no essential difference between the "heat on" and "heat off" surveys. Once heated, the tunnel is apparently slow to cool. This is illustrated by relatively minor changes in tunnel air temperature and tunnel "roof" temperature during the "heat off" survey in Figure 6.

2.2.2 First Experiment with AL Panels. October 1, 1965

Two panels were prepared for this experiment. One was striped with anisotropic liquid code numbers 5057, 5561, and 5964; the other

with code numbers 6268, 6670, 6873. It was thus possible with these two panels in combination to detect soil temperatures ranging from about 52° to 73°F (11° to 23°C). The heat source in the tunnel was the same as described in Section 2.2.1, i.e., 400 watt heat output from a tank of water stabilized for 48 hours at about 100°F.

Unfortunately, rain fell on the morning the test was scheduled and the panels as constructed were not suitable for use in the rain nor when water was lying on the ground. The windshield was not yet in place at that time. The rain stopped by noon and the panels were placed at various positions on the tunnel plot about 3:30 P.M. The soil temperatures at that time were such as to produce a color response on the #6873 stripe; none of the other stripes were active. The colors appeared in irregular areas, mostly green and blue, and sometimes small patches of orange were developed. The pattern of the colors shifted erratically as the panel was moved to various positions on the tunnel plot and sometimes these shifts occurred while the panel was allowed to remain untouched in one position. The outline of small stones, leaves, and small tufts of vegetation could be clearly distinguished in color when the "Mylar" base came in contact with these. The ground was wet, of course, but was relatively free of standing water and care was taken to avoid the few puddles present.

An effort was made to see whether some definite shift in color could be seen when the panel crossed the tunnel outline. Tests were made at both sides of the tunnel boundary and also at the rear boundary. Although shifts in color were observed as described above, these seemingly bore no relation to the tunnel boundaries. In some instances the colors indicated generally warmer temperatures on the soil over the tunnel; in other instances this was reversed and the colors indicated warmer temperatures on the soil beyond the tunnel outline. It was concluded that on wet soil at that time of day -- 3:30 P.M. -- local variations in soil surface temperatures were too erratic to permit detection of any small temperature difference that might be associated with the location of the heated tunnel.

A 24-hour temperature survey was recorded in conjunction with this experiment but is not included in the report since the panel tests were unsuccessful.

2.2.3 Second Experiment With AL Panels. October 21, 1965.

The second experiment with anisotropic liquid panels was carried out between 9:00 and 10:30 A.M. on a partly cloudy day which followed a week of clear days. The same two panels used in this test. The heat source in the tunnel had been allowed to remain "on" after the preceding test 20 days earlier, and as mentioned in Section 2.1.2, had been increased from 420 watts to 470 watts in order to offset the heat loss resulting from generally lower outdoor temperatures and thus maintain the water tank temperature fairly close to 100° F.

A temperature survey with thermocouples distributed according to Figure 1 was also included in this experiment. The final 10 hours of the survey are plotted in Figure 7. The shaded portion on this figure shows the temperatures that were recorded when the anisotropic liquid panels were being observed.

The temperature patterns represented in Figure 7 were in most respects similar to those which had existed during the corresponding time intervals in the earlier survey shown in Figure 5. As before, the median soil temperatures above the tunnel were about 1°F higher than the median soil temperatures outside the tunnel outline during the early morning hours. It is to be noted in this later survey, however, that the median temperatures began to fall very close together about 9:00 A.M. and were practically indistinguishable, one from another, between 9:20 and 10:20 A.M. when the panels were being observed. In spite of this apparent sameness of soil temperatures, one of the panels showed a color response that could be related to the location of the tunnel.

The panel which showed a color response was the one that was striped with anisotropic liquid code numbers #6268, and #6670, and #6873. A wide range of colored patterns was displayed since all three stripes became "active" during the test. The sketch in Figure 8, shows the general position of the panel and the color response of the three stripes at two different times, one near the beginning and the other at the end of the observation period.

It should be noted that only one panel is actually represented in Figure 8 although it may appear at first glance that two separate panels were placed end to end. The three-striped rectangle on the right hand or north side shows the position of this single panel in the windshield when the colored pattern was first observed at 9:30 A.M. The other three-striped rectangle on the left hand, or south, side represents the same panel moved to the other end of the windshield, which was done at 9:55 A.M. and again at 10:20 A.M.

The reversal in color pattern shown by the center stripe (#6670) in the figure is of particular interest. So far as this stripe is concerned, the pattern observed on the left hand side at 10:20 A.M. (also at 9:55 A.M.) was almost a mirror image of the pattern observed on the righthand side at 9:30 A.M. Also, since the anisotropic liquid responds to warmer temperatures by changing its color from black or brown through red and green to blue, the combined mirror images can be interpreted as showing generally higher temperatures above the tunnel and generally lower temperatures outside the tunnel. In making such an interpretation, of course, one is greatly helped by knowing where the heated tunnel was. There were also color variations on the other two stripes but it would be difficult to relate these color differences with any degree of certainty to the location of the heated tunnel.

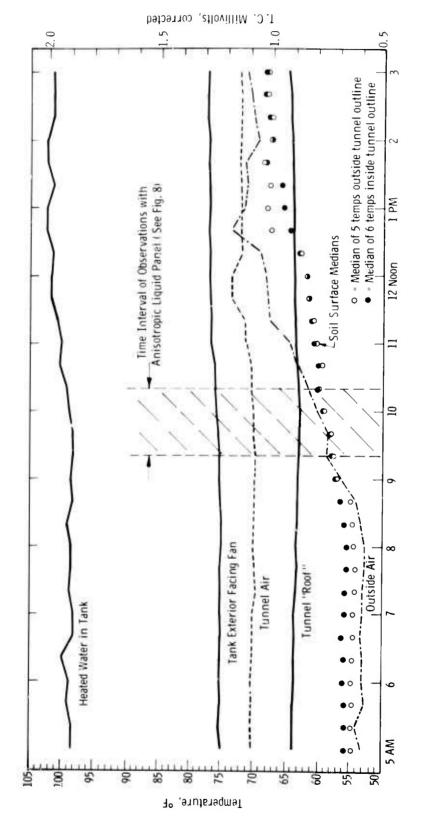
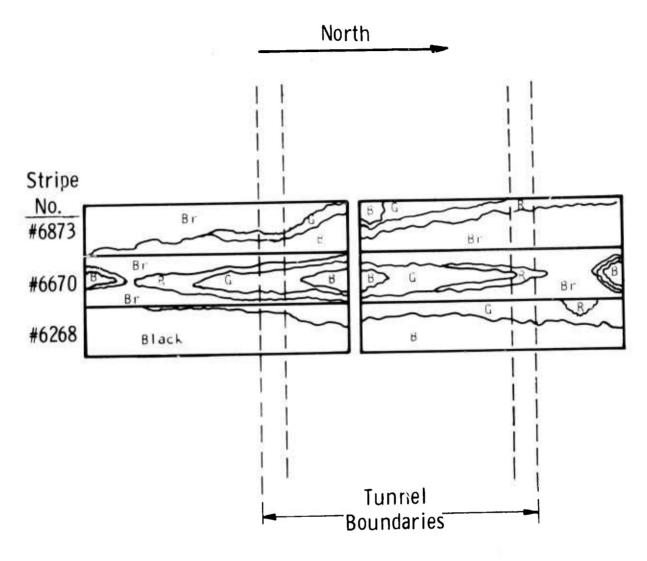


Fig. 7 —Final portion of temperature survey of 10/21/65. 470 Watts continuous fical in tunnel



B = Blue, G = Green, R = Red, and Br = Brown

Fig. 8—Colors observed on three-striped anisotropic liquid panel placed in windshield on tunnel plot on October 21, 1965.

North Side observed at 9:30 AM, South Side observed at 10:20 AM

It was observed during the course of the experiment that the panels as constructed were relatively slow in responding to changes in temperature. When the panel was shifted in the windshield it was always necessary to wait about five minutes before the color pattern could be considered representative of the new position. An even longer time was required at the start of the experiment when the panel was first placed in the windshield. In that case, the panel stripes were initially blue or black from being stored in an 80°F room. Gradually, other colors began to appear and after approximately 15 minutes, no further change was noticed. The relatively long time for the colors to become stabilized may be attributed to stored heat (from the 80°F room) in the aluminum frame and especially in the 0.125 inch thick"Plexiglas"cover.

To be complete, the experiment also included tests in which the panel was placed in direct contact with the soil. Various positions were tried inside, outside, and across the tunnel boundaries. As in the previous experiment (Section 2.2.2) mottled and variegated colored patterns appeared. These patterns seemingly indicated local temperature variations due to leaves, stones, etc., but bore no relation to the tunnel boundaries.

2.2.4 Third Experiment With AL Panels. November 4, 1965.

The third experiment was similar to the second experiment but was performed during the early morning hours before and including sunrise. The same thermocouple arrangement shown on Figure 1 was used and temperatures were recorded over a 24-hour period which included the time of tunnel detection studies and anisotropic liquid panels. Figure 9 shows the final 9 hours of this temperature survey plotted in the same manner as was used for the previous surveys. The shaded area as before indicates the previous surveys. The shaded area as before indicates the time interval occupied in carrying out the tests with anisotropic liquid panels.

In choosing the early morning hours for this experiment, it was hoped that the conditions when existing would allow the panels to respond more uniformly to the location of the heated tunnel. The previous year's program had shown rather clearly that random variations in soil temperature caused by naturally occurring influences such as sunlight and shadow would reach a minimum around sunrise and systematic temperature differences due to the heat source in the tunnel were then more likely to be discerned. The present series of experiments also tend to support this view. All of the temperature surveys indicate that the median temperature in the early morning hours on the soil surface above the heated tunnel were appreciably higher than the medians on the soil outside the tunnel. This was not always true at other times of day, as was briefly mentioned in discussing the second experiment in Section 2.2.3. A comparison of the 4:00 A.M. temperature surveys is shown on the next page in Table III.

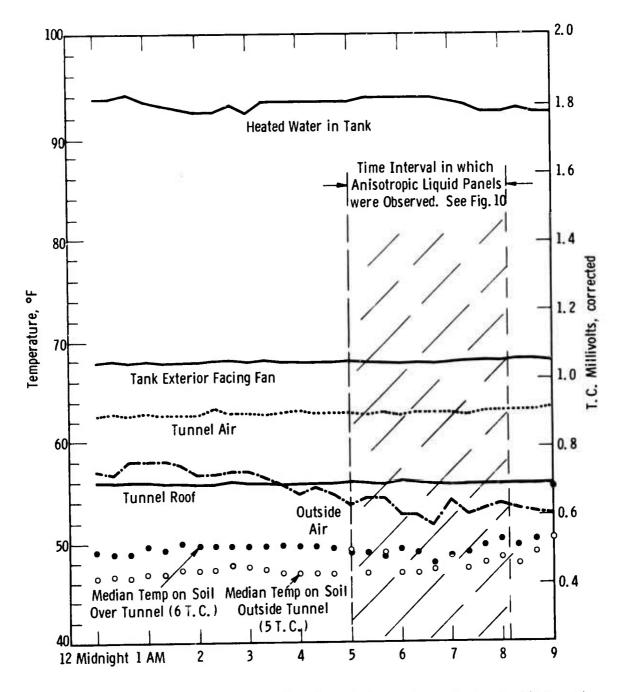


Fig. 9 —Final portion of temperature survey of 11/4/65. 470 Watts continuous heat in tunnel

Table III
4:00 A.M. Temperatures Recorded on Tunnel Plot in August, October, and November, 1965

Position of		Temperati	ures, ^O F	
Thermocouple	Aug. 12	Oct. 1	Oct. 21	Nov. 4
Median Soil Temp. Above Tunnel	61.0	64.5	55.8	50.3
Median Soil Temp. Outside Tunnel	60,5	63.5	54.5	47.3
Difference in Medians*	0,5	1.0	1.3	3.0
Outside Air Tunnel Air Tunnel Roof Water Tank	57.0 74.0 71.0 100.0	62.0 74.0 69.5 102.0	54.5 70.5 64.0 99.0	55.5 64.0 56.0 95.0

^{*}Subject to uncertainty of about 0.5°F.

It is interesting to note in this table that the temperature difference between the soil surface medians became progressively greater with the approach of colder weather. There is thus fairly good evidence that heat from the tunnel was actually affecting the soil surface temperatures and that the observed differences between the two medians were real and not merely the result of fortuitous placement of thermocouples on the tunnel plot. The widening spread between the medians was presumably due to steeper temperature gradients from the heated water tank to the overlying soil. This is also indicated by the parallel decrease in tunnel "roof" temperatures which dropped from 71°F on August 12 to 56°F on November 4 while the water tank temperature was held to 5° below its original 100°F setting.

The tests with the panels were begun at 4:15 A.M. and ended at 8:10 A.M. Observations were made by flashlight up to 6:30 A.M. at which time the dawn provided sufficient natural light to see the colors clearly. November 3 chanced to be a warmer than usual day for the time of year and the panel previously prepared from anisotropic liquid code numbers 5057, 5561, and 5964 (see Section 2.2.2) was found to be suitable for the test on the following morning.

The panel was observed inside and outside the windshield generally following the procedures already described in Section 2.2.3. Figure 10 is another diagram patterned after Figure 8 in that it represents the colors that were seen when the panel was placed in one end of the windshield and

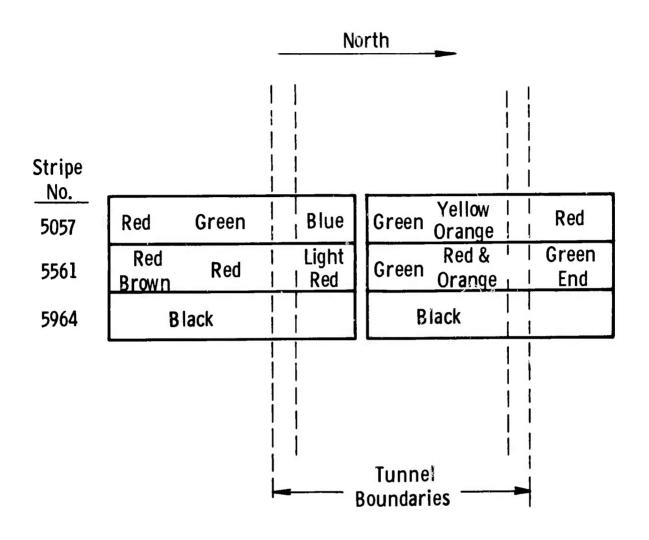


Fig.10-Colors observed on three-striped anisotropic liquid panel placed in windshield on tunnel plot on November 4, 1965
South side observed at 6:15 A. M.
North side observed at 6:30 A. M.

• later placed in the other end of the windshield. The first position shown, in the south end of the windshield at 6:15 A.M., was observed by flashlight. By the time (6:30 A.M.) the second position in the north end was observed, there was sufficient natural light to see without the flashlight. Other observations were made during the testing period, some by flashlight prior 6:15 A.M. and a larger number by natural light after 6:30 A.M. The color patterns were not always the same for the same position in the windshield but were generally in the direction expected, blues and greens where the anisotropic liquid films extended over the soil above the heated tunnel and yellows, red, and browns where the films extended outside the tunnel outline. Spurious effects were also noted at times; for example, a green or blue coloration where red or yellow should have appeared, or vice-versa. As a rule, however, the colors were fairly uniform in representing the warmer and cooler areas.

A photograph of the panel in the windshield was taken at 6:15 A.M. and again at 6:50 A.M. These two photographs are shown as a color contour sketch in Figure 10 earlier. The placing of the photographs corresponds to the rectangular panel diagrams in Figure 10; the left-hand photograph shows the coloration obtained in the south end of the windshield at 6:15 A.M., the right-hand photograph does the same for the coloration in the north end at 6:50 A.M. The broken white lines mark the position of the tunnel boundaries; the tunnel lies to the right of the line in the 6:15 A.M. photograph (south end) and to the left of the line in the 6:50 A.M. photograph (north end). The topmost stripe (#5057) is most easily interpreted in both instances. With the panel in the south end of the windshield at 6:15 A.M., this stripe is dark blue indicating a higher temperature in the short portion to the right of the broken line (i.e., above the heated tunnel) and grades very quickly through lighter blue to green and finally red indicating lower temperature as it extends to the left farther and farther away from the tunnel boundary. The definition at the boundary is not so sharp in the 6:50 A.M. photograph. However, here too the topmost stripe is blue indicating higher temperature at its extreme left-hand end where it lies over the heated tunnel. The colors from left to right then change gradually through mixed yellow and green as the stripe crosses the broken white line, and finally become reddish brown at the extreme righthand end farthest from the tunnel boundary.

As in all of the previous experiments, attempts were also made to locate the tunnel boundaries by placing the panel at various positions in direct contract with the ground. These attempts were again unsuccessful for the same reasons as before.

2.3 CONCLUSION

We believe that the feasibility of utilizing anisotropic liquid films for detecting a heat source simulating the presence of a human being hidden in a tunnel one foot below the soil surface has been demonstrated. There are, however, serious difficulties with spurious color effects produced by temperature differences arising from natural causes beyond the control of the experimenter or tester. At the present time it seems doubtful that the naturally occurring variations in soil surface temperatures can be eliminated to the extent necessary for practical detection of a human being hidden in a tunnel.

SECTION III

PACKAGING OF ANISOTROPIC LIQUIDS

The object of this portion of the investigation was to study materials that might be used for packaging anisotropic liquids. In the packaged form the anisotropic liquid film could be stored, transported or used to detect the temperature of the surface of an object.

For the best protection for the anisotropic liquid film against gaseous or solid contaminants, it should be sandwiched between two sheets of material. The material should be thin for easy heat transfer and transparent to permit visual observation of the AL color changes. Many plastic films have these properties in addition to being tough enough to permit the package to be handled repeatedly without breakage.

- 3.1. EXPERIMENTAL EFFECT ON COLOR RESPONSE
- 3.1.1. Packaging Materials in Contact with AL

Preliminary screening tests were made using 3" x .05" microscope slides. A coating of AL approximately 2 mils thick was placed on one slide. A cover slide was spaced 20 mils above the coating. The edges were taped to hold the slides together but not to exclude air. Three of the test cells are shown on the left side of Figure 11.

Table IV

Plastics Evaluated - Chemical Types

- 1. 'Mylar"- polyester
- 2. "Lexan"- polycarbonate
- 3. Polyvinylchloride plasticized
- "Celcon"- Acetal polymer
- 5. "Tedlar"- polyvinylfluoride
- 6. Nylon, Zytel 101 polyamide 7. "Capran," Nylon 6 polyamide
- 8. "Surlyn A"- Inomer
- 9. "Aclar 33-C"- trifluorochloroethylene
- 10. Cellulose acetate mono
- ll. Cellulose acetate tri
- 12. "Kynar"- polyvinylidene fluoride
- 13. Polystyrene
- 14. "Lucite"- methylmethacrylate
- 15. "Teflon 100 FEP"- polyfluoroethylene propylene
- 16. "Dynel"- copolymer of acrylonitrile and polyvinichloride
- 17. ABS polyacrylonitrile-butadiene-styrene

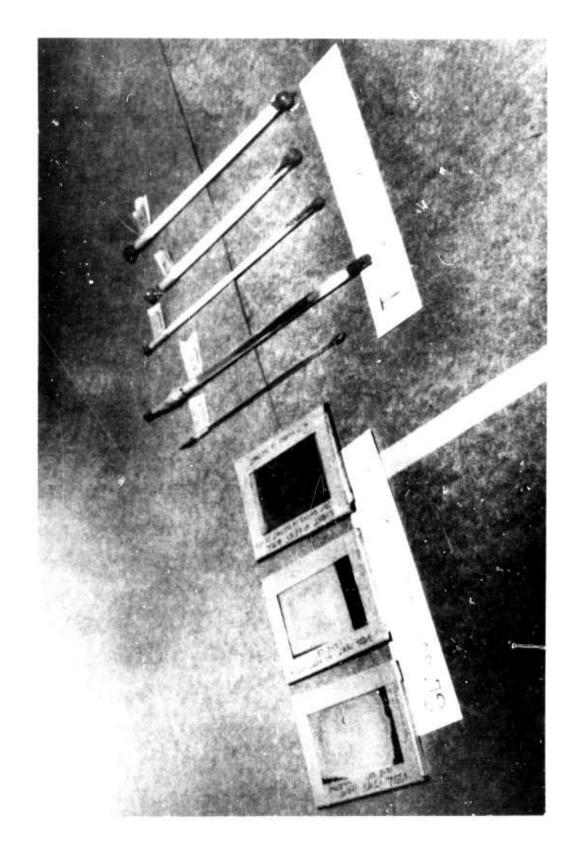


Figure 11. Anisotropic Liquid Test Specimens

Table V

Depression in Color Appearance Temperature for AL #6873* Aged at Room Temperature in Contact With Various Materials

AL #6873 in contact with	(days)	(² F)	(days)	(^O F)	(days)	$(^{\circ}_{F})$	(days)	$(^{O}_{\mathbf{F}})$	(days)	(°F)	(days)	(°F)
Glass, in room light	9	7	3	9	143	80	64	6	92	15	•	
Glass, in dark	9	6	8	4	22	9	9	80	72	6	100	15
Glass, in dark, 2 mil film	3	7	17	7	50	77	917	9	8		87	13
Glass, in dark, 4 mil film	(٣)	-	1		5¢	9	1.4	7	70	8	8	13
Glass, in dark, 6 mil film	٣	1	23	7	5 4	9	ß	80	61	6	88	13
Polyethylene	7	-3	5.	9	•	1				. 1		
ABS	6	1,	11	9	•	1	;	•	•		•	•
Lucite	10	-7	14	9	•	•	•	•	•			,
Polystyrene	7	77	10	9	•	ı	•		•		•	,
Polycarbonate (Cr-39)	7	*	ίl	9	•	1	•	•	•		•	
Polypropylene	80	-1	12	2		•	•	•	,			,
Lexan	22	0	•		28	4	**	9	去	10	•	ı
Celcon	-#	Ŋ	22	4	28	9	42	υО	55	13	•	•
Tedlar	-3	7		•	28	7	77	9	55	10	•	•
2ytel 101	9	Ŋ	21	4	27	y	04	10	55	15	1	
Capran	5	5	59	7	R	9	2	8	78	6	96	1,4
Surlyn-A	4	5	30	N		ī	55	2	69	8	96	رد
Aclar 33-C	2	2	59	5		,	84	10	55	12		•
Kyna r	7	8	7	6	21	13	,	ı		r	•	,
Cellulose Acetate, Mono	4	15	•		•		ı		•	1	•	•
Cellulose Acetate, Tri	-#	12	•	1	•	ı	•	,	•		•	ı
Polyvinylchloride	- ≠	18	•		•	ī	•		ı	•	•	
Mylar	10	2	1	•	35	7	84	9	55	7	19	14

* Recipe #6873 original color appearance temperature blue at 70°F ** Plasticized

Samples of the plastics listed in Tables IV and V were obtained in sheet form which ranged from 1/32 to 1/8" in thickness or in film form up to 2 mils. Pockets 1/2" in diameter were milled into the top surface of the sheet materials to form thin bottomed cells. The cell bottoms were 15 to 20 mils thick. The solution of AL (recipe No. 6873 in solvent) was added to the plastic surface dropwise until after evaporation a 2 mil film of AL was deposited on the thin plastic surface. Recipe No. 6873 was a 10% solution of 63% oleyl cholesterol carbonate - 28% cholesterol nonanoate - 9% cholesterol propionate. Petroleum ether and chloroform 1:3 was used to dissolve the AL.

3.1.2. Testing Procedure

A controlled tempers ture surface was formed by the top surface of a 3" x 2" copper box from 1/32" thick sheet. After the preliminary test a 3" x 4" surface with better flatness was constructed. The boxes were fitted with an inlet and outlet tube which permitted water from a constant temperature both to be circulated through them.

The temperature of the circulating water was adjusted to a temperature slightly above the temperature at which color could be observed in the AL film.

The glass or plastic base on which the AL film was spread was laid on top of the controlled temperature surface. The bottom of the specimen has been blackened by a coating of black spray enamel ("Krylon No. 1601 or No. 1602) to provide a better background against which color changes in the AL film could be observed.

The temperature of the circulating water was reduced in one degree F steps allowing 3 minutes for the surface and specimen to come to equilibrium at each temperature. The temperature at which color (blue) first appeared was recorded as the color appearance temperature. The temperature was reduced in this manner until the complete color range of the AL film was spanned and color at the red end of the range disappeared. The color range of some of the specimens was determined as the temperature range from the first appearance of blue until the disappearance of all green. These points were more easily observed. This procedure was used for the Ring specimens and the glass slides run in the later part of the program. Color observations were made in the laboratory fluorescent lights as it was reflected from the AL film on a glass or plastic background. A piece of 3" diameter tubing blackened on the inside was directed at the sample at an angle of 30 degrees from the vertical. The observations, after this refinement, were all made at the same angle to the specimen. As an additional aid to observation a box was constructed around the specimen and controlled temperature plate. The box was blackened on the inside to cut down stray reflected light which made color observations difficult. Other light sources were used such as daylight and incandescent bulbs to observe any influence they had on the color observations.

A diagram of box and tube is shown in Figure 12.

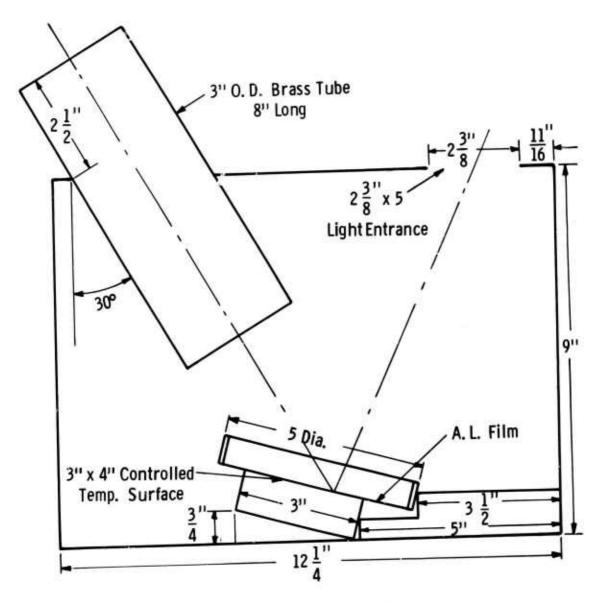


Fig. 12-Diagram of color change viewing box

3.1.3. Second Aging Test: AL (10-28-65 Batch)

A sample of anisotropic liquid designated 10-28-65 batch was procured. This material was not in solution. The viscous liquid-crystalline material was composed of 70 parts oleyl cholesterol carbonate, 30 parts cholesterol nonanoate and 6 parts cholesterol benzoate. The material was clear at temperatures above 80°F and turned from blue to green to rust to become clear again as the temperature was dropped to 75 to 76°F. The AL sample was stored in a brown glass bottle and in a dark drawer to prevent as much as possible any deterioration due to ultraviolet light from the fluorescent light fixtures in the laboratory.

3.1.4. Specimens for Aging Tests

Brass tubing (1/8" wall) was obtained in two sizes that could be fit one inside the other by machining the outside of the smaller. The clearance between the two tube sections could be varied to accommodate the thickness of the plastic film being stretched across the opening to hold the anisotropic liquid. The wall thickness of each ring was reduced to 1/16" for a 1/4" distance in order to form a groove 1/8" wide by 1/4" deep when the two 1" long sections were fit together. The purpose of this groove was to provide a space into which an epoxy resin could be poured after the films had been stretched over and held in the clearance between the rings. This potting served to seal the edges and hold the film in place. The inside diameter of the smaller ring was 4-1/2". See Ring No. 2, Figure 13.

Ring No. 1 was fitted with a sandwich containing the new 10-28-65 anisotropic liquid sample. A sheet of 0.5 mil "Mylar" was coated with a 1 g of anisotropic liquid over a 7" x 7" area. A second sheet of "Mylar" was laid over this and rolled to remove air bubbles. The two sheets were stretched over the rings by forcing the outer ring over the film laid across the inner ring. The anisotropic liquid film would be less than 1 mil in thickness.

Ring No. 2 was fitted with a sandwich made from two layers of 2 mil "Mylar". The surface of the "Mylar" was washed with water and a detergent, rinsed, and air dried before the anisotropic liquid was applied to its surface. The sandwich was evacuated and free air worked out before the brass rings were put in place. The edges of both rings No. 1 and No. 2 were sealed with epoxy resin. After the resin had set, the color range of the specimens was determined in the usual manner on a cooled plate. The temperature at which the purple color first appeared and the temperature at which the green disappeared were recorded as the temperature range of the panel. Both rings are being stored under cover to eliminate the effect of UV light deterioration.

Ring No. 3 was made by the procedure described above. Two layers of 3 mil "Surlyn A" plastic film with the AL batch (10-28-65) between them formed the sandwich for this specimen. The back (contact side) was sprayed with a light layer of "Krylon" black spray enamel 1601. Specimen prepared in a clean bench.

Ring No. 4, Figure 13, was made by stretching a sheet of 3 mil "Surlyn A" over a 4-1/2" ID x 1" wide brass ring. A 3/8" wide outer ring was forced over to clamp the film in place. The AL (10-28-65) was applied to the inside surface of the "Surlyn A" and spread to form a 1 mil film. A sheet of 1/2 mil "Mylar" was stretched over the top of the ring and held in place by a 3/8" wide ring forced down over the top. The "Mylar" acted as a cover to prevent contamination. The specimen was prepared in a clean bench. The bottom surface of the "Surlyn A" was sprayed with "Krylon" black enamel. The air above the AL layer was displaced by nitrogen which was introduced through a 1/16" hole in the side of the brass ring between the two clamp rings. A hole in the opposite side permitted gas and air to escape. Nitrogen was passed in at a slow rate for 15 minutes. Inlet and outlet holes were then sealed with adhesive tape.

Ring No. 5 was constructed according to the procedure described above. The specimen area was formed by two layers of "Capran" film, type 80, 2 mil, with a 1 mil layer of AL (10-28-65 batch) between. The AL was applied to the surface of the film on a small area at the center. The top film was applied and the sandwich clamped between the brass rings. The film sandwich was laid on a smooth surface. Pressure was applied to the top surface by a smooth metal bar to spread the AL evenly over the circular area and work any air bubbles to the perimeter of the sandwich. The work was all done in a controlled environment.

Ring No. 6 was prepared as a duplicate of No. 4, except that the air over the AL was not displaced by nitrogen.

Ring No. 7 was constructed in the same manner as No. 5. The film used was FEP "Teflon" 100, 1 mil, 2 layers. The AL layer between was 1 mil thick and from the 10/28/65 batch.

Ring No. 8 was a 1 mil film of AL. (10-28-65) was sandwiched between two layers of 2 mil polycarbonate film. The sandwich was installed on the face of a brass ring in the same manner as described above.

Rings No. 9, 10 and 11 See pressure package specimens.

Ring No. 12 was a 1 mil film of AL. (10-28-65) was sandwiched between two layers of 1 mil "Surlyn A." The "Surlyn A" was cleaned with methylethyl ketone, washed with detergent and rinsed three times in cold water. The film was dried in a clean bench prior to being used to contact with AL. The sandwich was clamped between two 5" diameter embroidery hoops.



Figure 13. Anisotropic Liquid Test Specimens

Ring No. 13 was assembled in the same manner as No. 12 except that the "Surlyn A" film was not treated to remove any anti-friction material applied to the surface by the supplied during rolling.

Ring specimen No. 14 was two pieces of 1 mil "Surlyn A" washed with MEKetone; detergent and water; rinsed three times in tap water and dried in the clean bench. A 5" diameter disc of "Dynel" veil, "Pellon" 5362, was cut. A thin film of anisotropic liquid (AL - 10-28-65) was spread on one of the "Surlyn A" sheets approximately the size of the "Dynel" disc. The 0.005" thick "Dynel" disc was pressed into the AL by a clean steel blade and AL was spread on top of the "Dynel" to fill the openings. The second "Surlyn A" sheet was put on top of the coated "Dynel." The sandwich was rolled from the center out to spread the AL and work air pockets to the edge of the "Dynel" area. The sandwich was stretched over a 5" diameter embroidery hoop and clamped.

Ring specimen No. 15 was made in the same manner as No. 14 except 0.004" thick heat cleaned glass cloth style 112 was substituted for the "Dynel."

Results of the aging tests are presented in Table VI.

3.1.5. Third Aging Test: AL Coated on Various Porous Materials

A series of ten samples were prepared with the following construction:

- 1. A 6 mil layer of AL was spread on the surface of a 3" x 2" x 0.050" mil microscope slide. A second slide was used as a cover but spaced 25 mils above the first so that there was no contact between the top slide and the AL (10-28-65 batch) film. The edges were taped to hold the top glass but not to exclude air. Three sample cells are shown on the left of Figure 11.
- 2. A 6 mil layer of AL (10-28-65 batch) was spread on the surface of a 3" x 2" microscope slide. A second slide was pressed into the AL layer to form a contact between the glass and the AL. The edges were taped to hold the top glass in place.
- 3. A thin coating of AL (10-28-65 batch) was spread on a 3" x 2" microscope slide. A 1" x 2" piece of absorbent paper 4 mils thick was pressed into the AL. Additional AL was then spread over the top of the paper. A second slide was pressed lightly onto thetop and the edges were taped to hold the glass in position.
- 4. A mixture of AL (10-28-65 batch) and 3% "Cabosil" was made. The paste formed was spread 6 mils thick on a 3" x 2" microscope slide. The paste spread more evenly than the unfilled AL. The top glass plate was spaced 25 mils above the bottom plate so that no contact was made between it and the AL film.

Table VI

Depression of Color Appearance Temperature For (AL 10-28-65 Eatch) Aged in Contact with Various Plastics

ø)	O F					2 ⁸				8	0	0	7	7 1
perature	Days	15	15(141	130	117	130	117		8	79	75	57	57
е Теп	O.	7	ω	5	a	4	7	ω		m	0	0	0	П
Lowering of Color Appearance Temperature	Days	145	140	131	120	104	120	104		70	54	Ţ.	78	24
olor	0 F4	9	ω	4	0	7	0	7		n	0	С	ľ	ı
ring of C	Days	%	も	8	20	55	20	55		21	7	5	ľ	ı
	O _E	m	4	m	0	Q	0	0		t	ı	t	ı	
Time and	Days	77	69	57	45	30	45	30		ı	ı	ı	ı	ı
Aging	C	1	a	1	7	t	ı	ı		ı	ı	1	ľ	ı
	Days	37	35	22	11	ı	ı	•		ı	1	•	ı	1
Material in Contact	With AL	Mylar	Mylar	Surlyn-A	Surlyn-A ²	Capran	Surlyn-A ³	FEP Teflon	100	Polycarbonate	Surlyn-A		Surlyn-A, Dynel	Surlyn-A, Glass'
Ring	No.	٦	ณ	3	7	5	9	7		ω	75	13	14	₁ ,

Sandwich of AL Between two Layers of Film

Blue color appears in spots; color not uniform over test

> Free AL Film, Air Above Displaced By Nitrogen a

Free AL Film, in Contact with Air, Covered

Surlyn-A, 1 Mil, Surface Cleaned

Surlyn-A, 1 Mil, Surface as received S

on Dynel Veil Between Girlyn A Films AL

on 112 Glass Cloth Similar to Sample 9 Table IV

AL

area; redistribution of AL between films did not improve color response

- 5. A thin layer of AL (10-28-65 batch) was spread on a 3" x 2" microscope slide. A 1" x 2" x 6 mil piece of surface mat (random fiberglass mat normally used to surface glass reinforced structures) was laid into the AL; then AL was spread over the top to fill the voids of the glass mat. The top glass slide was pressed against the AL-may layer. The edges were taped to hold the glass plates in place.
- 6. A duplicate of No. 5 except that the glass mat was heated in a flame to burn off any organic binder that may have been on the glass fibers.
- 7. A mixture of 90 parts Berkley Special silica sand and 10 parts AL (10-28-65 batch) was used to coat the surface of a 3" x 2" microscope slide. A second slide was pressed into the AL layer to form a film 16 mils thick. The edges were taped to hold the glass slides in position.
- 8. A duplicate of No. 7 except the top slide was not in contact with the AL film layer.
- 9. A 1" x 2" layer of 4 mil thick ll2 woven glass cloth, heat cleaned as received, was coated with AL (10-28-65 batch) as in samples 5 and 6. The top glass was placed in contact with the AL film. The edges were taped to hold the glass plates in position.
- 10. A duplicate of No. 9 except that the glass cloth was heated in a flame to drive off any volatile binder or moisture that might have remained on the glass.

Results of aging tests are presented in Table VII.

- 3.2. EXPERIMENTAL PACKAGING EFFECTS
- 3.2.1. Influence of AL film Thickness on Color Change

Film Thickness Specimens - Films of AL (10-28-65) were made between two 3" x 2" microscope slides. Spacers of 0.5, 2.0, 10.0, 30.0 and 60.0 mil thickness were used to adjust the spacing between the upper and lower slides. The edges were taped to hold the slides in place. The rate of sample temperature rise when heated from 70°F to an 82°F plate was measured. The time taken for the sample to loose its green color as it heated was also recorded. These data are presented in Table VIII.

Table VII

Anisotropic Liquid (AL 10-28-65) Aged Between Glass Plates and Crated on Various Materials

Days Aging in Dark at R.T. and Drop in Color Appearance Temp., ^O F, From Original of 80 ^O F	OF.	3	1	7	+17	0	0	7	m	0	0
or Appe F	Days	115	115	115		115	114	114	115	114	114
7 Col	o _F	H	7	7	+17	0	0	7	r d	0	0
Drop in	Days	105	105	105		105	104	104	105	104	104
and	o FI	7	7	7	+17	0	0	П	Н	0	0
R.T. From	Days	83	83	83	83 4	83	88	88	83	8	88
irk at	OF	1	Н	7	+18	0	0	0	П	0	0
in De Temp	Days	55	55	55	55	55	75	54	.55	47	75
Aging	O _F	7	7	0	+197	0	0	0	1	0	0
Days	Days	30	8	8	8	8	59	59	30	59	59
Top Glass Plate in Contact With AL		No	Yes	Yes	No	Yes	Yes	Yes	No	Yes	Yes
Materials Coated With AL		Glass	Glass	Crepe Paper	Cabosil M-5	Surface Mat	Surface Mat	Silica Sand	Silica Sand	Glass Cloth	Glass Cloth
AL Film Thickness, (Mils)		9	9	7	9	9	9	16	16	21	6
Sample No.		1	ત	m	4	2	9	7	ω	6	10

Microscope Slides 3" x 2" x 0.05"

Surface Mat, Glass, Random Fiber, 6 Mils Thick, as received

3 Surface Mat, Same as Above Heat Cleaned

Berkley Special Sand

5 Glass Cloth, 112, Heat Cleaned, J. P. Stevens Co.

5 Same as 5, Heat Cleaned in Lab

Only Material Mixed With AL That Raised Temp. and Expanded Color Range

Table VIII

Rate of Color Change of Anisotropic Liquid
Samples Heated from One Side

AL Film Thickness Mils	Temp. of Specimen	Temp. of Heating Plate, OF	Time for Green Color to Disappear Secs.
0.5	70	82	42
2.0	70	82	62
10.0	70	82	72
30.0	70	82	91*
60.0	70	82	126

^{*}Color disappearance not as well defined as in thinner films.

Green color not distinct, the appearance was an opaque greenish white in this and greater thickness.

3.2.2 Tubular Packages for AL Films

A 6" long piece of 1/8" ID glass tubing was vacuum filled with AL (10-28-65 batch). The ends were plugged. One-half of the exterior surface was painted black. The color response temperature was checked in a water bath in both descending and ascending directions.

A 6" length of 0.080" OD glass tubing was vacuum filled with AL (10-28-65 batch). A piece of 0.032" OD glass tubing filled with black paint (ends sealed) was inserted into the larger tube to form a centrally located black background. The color response range of this system was checked in a water bath.

Other Tubular Specimens - A number of "Pyrex" glass tubes were obtained to make specimens of the cross sections shown in Table IX and Figure 11. The outer tube was partially filled with AL (10-28-65). The center tube was, in most cases, filled with black paint. The ends were either closed with epoxy resin or heat sealed. A better center tube was made with black iron oxide dust. The ends were heat sealed with no volatile materials to interfere. The black center tube was inserted in the center of the tube containing the AL. The tube was centered and the ends sealed with epoxy resin compound or sealing wax. The samples were taken from a water bath at 70°F to one at 80°F. The time for the sample to assume its clear color was measured.

Table IX

Rate of Color Change for Anisotropic Liquid
Samples Surrounded by Heat

AL Film Thickness Mils	Time From Opaque -75°F to Clear 80°F	Relative Size of Glass Tubing Cross Sections
3.5 ¹	2	
5.5	4	
16.0	4	
22.0	13	
29.0	7	
54.0	9	
57.0	7	

^{1.} Shaded portion represents AL. Center of inner tube filled with black paint or black iron oxide powder. AL 10-28-65 sample.

3.2.3. Pressure Package for AL Films

Two 5" diameter by 1" high boxes were made from 1/8" thick "Lucite." The top of each box was a disk of 1/8" "Lucite." The bottom was open. One of these boxes designated Ring No. 9 was fitted across the open face with a sandwich composed of 2 layers of 3 mil "Surlyn A" with a 1 mil layer of AL (10-28-65) between. The sandwich was clamped to the body by a "Lucite" clamp ring 3/8" wide. A 1/4" OD "Lucite" tube mounted in the side of the box permitted air to be pumped in from an atomizer bulb. The flexible film sandwich could be made to conform to the contours of a slightly irregular surface by applying pressure inside the box as it was held against the surface. The second box (Ring No. 10) was fitted with a 5" ID, 7" OD flat flange ring cut from 1/8" "Lucite" and adhered to the box at the open face side. A second ring of the same size was cut to act as a clamp ring for the AL sandwich to be applied to the bottom of the package after both holes had been drilled through it and the flange. Enough slack was left in the sandwich to permit it to bulge slightly beyond the plane of the lower clamp ring where internal pressure was applied to the box. A 1/4" OD "Lucite" tube was connected to the side of the box to which an atomizer bulb could be attached. A 1/32" rubber gasket was used to seal the flange area against loss of air. This package was designated Ring No. 10. The package designated as Ring No. 11 was a duplicate of No. 10 except that it was a 5" x 5" x 1" square box to demonstrate the ability of the "Surlyn A" sandwich to deform satisfactorily in both round and square configurations. They are shown in Figure 14. Figure 15 shows a larger square (12" x 12") pressure package being used against an uneven surface.

3.2.4. Removal of Wrinkles from "Surlyn A"

A 3 foot long piece was cut from the 1 mil and 3 mil 27" wide rolls of "Surlyn A." The materials were wound on to a 1-1/2" diameter cardboard tube while heat was being supplied from heat lamps placed so that the entire width of the sheet was heated as it passed on to the roll. A five pound load in the form of two steel bars clamped across the free end of the sheet supplied a tension force to the sheet during rolling.

A kraft paper sheet was introduced between the plies of "Surlyn A." It was wrapped in order to provide the slightly tacky surface of the warm "Surlyn A" with a chance to slip on itself, roll straight and eliminate the wrinkles which have been present in all the rolls of "Surlyn A" that have been examined.

3.2.5. Vertical Flow of AL Film

A 1 mil film of AL was spread between two sheets of "Surlyn A." The edges were heat sealed to form an envelope 6" x 8". The package was hung at room temperature by a corner to observe the tendency for the AL to flow.



Figure 14. Pressure Package for Anisotropic Liquids

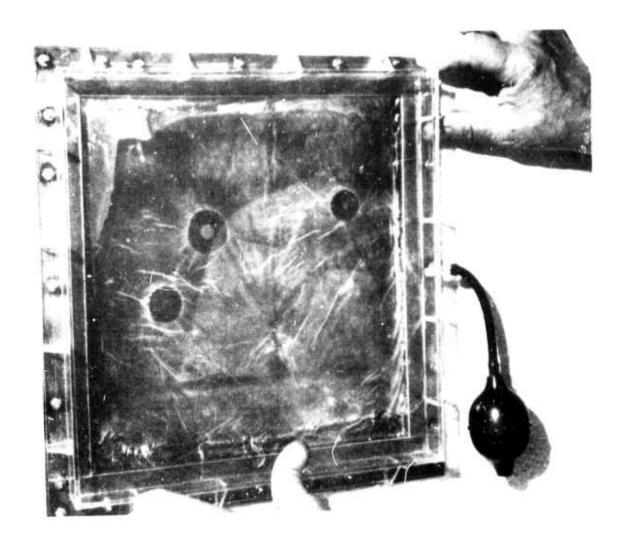


Figure 15. Large Pressure Package for Anisotropic Liquids

Similar samples were hung in a 50° C oven for a higher temperature test.

No flow was observed in the AL (1 mil) film sandwiched between two plies of "Surlyn A" that was suspended vertically at room temperature for 12 days or for 48 hours at 50°C.

Very slight flow was noted in one of the samples that have been hanging in vertical position at room temperature for 45 days.

Sandwich type samples similar to those above but containing a sheet of 112 glass cloth coated with AL (10-28-65) were suspended by a corner at room temperature and at 50°C. The glass cloth was not clamped. No flow of the AL or movement of the glass cloth was noted after 4 days at 50°C. At room temperature there has been no flow or movement of cloth after 42 days.

3.2.6 Sealing of "Surlyn A" Film

Heat Sealing - The continuous belt heat sealer in the laboratory was used to make seals in both 1 and 3 mil "Surlyn A". The temperature of the hot zone of the sealer was $290 - 330^{\circ}$ F.

Seals were made in uncoated film and in film coated with AL.

Impulse Sealing - An "Audion" impulse sealer was used to make successful seals in both coated and uncoated "Surlyn A".

Ultrasonic Power Sealer - The ultrasonic sealer was not successful in sealing "Surlyn A". Experiments made by Branson Laboratories indicated that this method was not applicable.

Dielectric Heat Sealer - Experiments on sealing by this method were not successful. A DuPont representative also stated that this method was not applicable.

3.2.7 Vacuum Forming of AL Packages

Standard production type vacuum forming equipment was used. The plastic sheet was heated from above until it became soft enough to sag. The clamping frame was lowered as vacuum was drawn in the chamber containing a male mold form. The external pressure forced the softened film around the form. The softened film stretched slightly to permit close duplication of all the contours of the form.

The forms consisted of (1) a wood block 12" x 12" x 3/4" with edges and corners rounded and a 10 degree draft on the four vertical sides; (2) four wood blocks 5-1/2" x 5-1/2" x 3/4" with the same draft as before. These could be used for forming a cluster of four pieces or single 5-1/2" x 3/4" trays; (3) an aluminum block 4-1/2" x 3-1/2" x 1/2".

After the film had been drawn around any of these forms and cooled, a flat bottom tray with flash perpendicular to the top of the sides was produced. The flash could be cut back to any desired width.

The materials used to form trays were:

- 1. 30 mil "Forticel", Celanese Formula 406.
- 2. 10 mil "Forticel".
- 3. 1, 3, and 15 mil "Surlyn A".
- 4. 10 mil "Forticel" and 1 mil "Surlyn A" were molded simultaneously with the Surlyn A" forming the inside layer.
- 5. 15 mil "Surlyn A" mold over the "Surlyn A".

Trays molded from "Forticel" (5-1/2" x 5-1/2" x 3/4") were coated on the inside of the bottom with a thin film of a permanently tacky material. "Daratak" 74-L, an acrylic emulsion and uncatalyzed "Epon" 828 were used. A sandwich of AL between two layers of 1 mil "Surlyn A" was adhered to the coated "Forticel" surface. A coating of black (flat) enamel was sprayed on the outer (contact) surface of the tray to form the usual black reflecting surface for viewing AL color changes.

3.3 DISCUSSION

Anisotropic liquids (AL) change color with temperature when viewed by light reflected from a dark background upon which a thin film of the material is spread. The temperature range through which color changes take place is a function of the AL recipe used. The film is clear above and below the temperature range. The film passes in stages from blue through mixtures of green and rust color as the temperature is lowered through the color range of the specific formulation. Temperature differences at the surface of an area may be detected by observing the color changes in an AL film placed on the surface if the color range of the AL is adjusted properly. If the AL is coated on some thin background the film may be used repeatedly and transported from place to place.

The package in which the AL is placed for use in surface temperature surveys must be made of materials that will not alter the color transition temperature of the system appreciably after a reasonable storage time.

The preliminary screening test was made to determine the effect that the packaging material would have on the color transition temperature of the AL. A number of plastics were chosen to include many chemical structures. The materials chosen are listed in Table V. The AL films were tested on glass for comparison with plastics to observe any differences in color reaction on in inorganic and organic surfaces. The AL used in all these tests was No. 6873 in solution.

After a few days of testing it was apparent that there was a gradual drop in the top temperature at which color appeared in the AL film. Table I shows the rate at which the decrease progressed. In the early part of the test the "Mylar", "Capran" and "Surlyn A" specimens inhibited the color appearance drop more than the others. However, "Surlyn A" showed greater inhibiting action than any of the others as the aging test continued. The color appearance drop at the blue end of the color band was also reflected in the green and rust colors that appeared at slightly lower temperatures. The width of the color band remained substantially constant while the color appearance temperatures within the band gradually dropped.

The AL specimens (Table V) that were prepared on glass exhibited the same changes that had been observed on plastics. Those films that were stored in the dark aged more slowly than those exposed to the laboratory fluorescent lights.

The factors that might influence the drop in color appearance temperature were light, solvent and the polymeric packaging material. No definite drop in color appearance temperature was designated as a limit to the usefulness of an AL film but drops of 8 to 15°F would seem to be beyond a practical limit.

A new sample of AL was obtained that was designated as AL (10-28-65 batch). No solvent was added to this material. The AL was stored in a brown glass bottle in a drawer away from any exposure to ultraviolet light.

A series of samples in the form of packages for AL were made. These samples are described in the experimental section as Ring samples, Table VI, Figure 14. The combined refinements in construction and storage of these samples were responsible for a retarding of the drop in color appearance temperature for the AL--plastic systems that were observed. For example, the "Capran" (Table IV) specimen shows a 9°F drop in 78 days while "Capran" (Table VI) at 104 days shows a 4°F drop. Also the order of resistance to drop for "Surlyn A", "Capran" and "Mylar" is the same in both Tables IV and VI even though the magnitudes are different. The degree to which each factor, light, solvent, base material, purity of the AL and freedom from contamination contributes to the retardation of the color appearance drop cannot be fully estimated here, but the plastic is certainly an important contributing factor. The other factors should be controlled to the greatest extent necessary for maintaining the color change sensitivity needed for the application.

Since the intended application for the packaged AL involved observation of color changes visually, all color tests on specimens were made visually. The viewing box shown in Figure 12 was found to be helpful in duplicating more closely the color readings made over an extended period. The important feature is that viewing conditions and angle are the same for each test. Light sources could also be changed when the viewing box was used. The regular laboratory fluorescent fixture light

was replaced by that from a (1) daylight incandescent bulb, (2) frosted incandescent bulb. The observations made with the laboratory lighting and (1) were essentially the same. When (2) was used the blue color appearance was more difficult to distinguish but the green band of color was much more visible than with the other sources.

A series of specimens were made to show the effect of AL aging when it was coated on various materials that could be used as spacers between two sheets of plastic film. Examples of these are shown at the left of Figure 11. The purpose for these materials was to insure an even thickness film over the area of the AL package. The materials and the results of the aging test are listed in Table IX. The only material that had an adverse effect on the AL was "Cabosil" M-5. Instead of the normal 4 to 5°F color band width usually experienced with AL (10-28-65) the blue color in the "Cabosil" mixture persisted for as much as 19°F higher. The green color band however was raised only about 1°F.

The presence of air above these films 1, 4 and 8, Table VII did not seem to influence the color appearance drop. This is also shown in Table VI, numbers 4 and 6 where the air in the package was displaced by nitrogen.

The data listed in Table VIII indicate that the most effective film thickness for use in indicating good color throughout the recipe (10-28-65) temperature range was 10 mils.

Thicker films assumed an opaque green-white color instead of the vivid green of the thinner film before going over to the blue range. The color reaction time was a function of film thickness. The plate temperature in this test was set at $82^{\circ}F$ which was just above the normal $80^{\circ}F$ clear point for the material.

The tubular samples showed much less difference in reaction time for the thickness of film examined. Tubes could not be obtained to make a series with all the outside tubes the same diameter. Some of the slight time differences, indicated in the test data, could be due to this fact. The use of a black center tube permits better observation of the AL color changes than in a tube filled with AL and blackened on parts of the exterior surface away from the observer.

The packages that may be pressurized with air as shown on Figures 14 and 15 permit the flexible plastic-AL sandwich to be applied intimately to an uneven surface and observed through a transparent plastic top. Intimate contact is difficult to acquire on a vertical surface. Figure 15 shows the draw down into slight depressions on a vertical surface. The amount of formability would depend on the amount of slack built into the flexible sheet. The low pressure at which this package operates require that the gasketing be leak proof. The gasketing arrangement shown as the flange and ring gave the most satisfactory seal. Ring No. 9 in which a belt clamp was used did not seal well and the clamp ring had a tendency to creep. The only advantage of this arrangement was that it

placed the AL sandwich 1/8" closer to the bottom of the package at the edge. Iarger packages could easily compensate for this as the percentage of area lost at the edge was decreased.

Wrinkles may be removed from the "Surlyn A" by wrapping the material under tension with enough heat applied to soften the film slightly. The use of a kraft paper inner liner is necessary to maintain flatness. "Surlyn A" is normally dusted with powdered starch while being rolled in order to provide slippage between plies. Any rolls that have been examined have had wrinkles. For general use in blister packaging this is not serious because the material becomes heated and is stretched during vacuum forming. Inquires made indicate that a supplier would have to be equipped specifically for this operation. The quantity of material ordered would also have a bearing on the price. It would be desirable to have undusted "Surlyn A". Perhaps a supplier could work out a method in which elaborate winding equipment would not be necessary to procure reasonable quantities of material wrapped with an inner liner sheet.

The films of AL between "Surlyn A" that have been suspended vertically have shown slight flow in only one case after 45 days at room temperature. See information in experimental section on this subject.

Sealing of "Surlyn A" - "Surlyn A" may be heat sealed with a coating of AL in between at the sealed area. Successful seals have been made at 290 - 330°F. The impulse sealing method seemed to make the most satisfactory seals. An electrical impulse is put through a thin ribbon heater on the surface of the sealing jaw. The ribbon cools very quickly allowing the seal to cool before it is released from the jaw. Neither dielectric heating nor ultrasonic power sealing has been used successfully with "Surlyn A".

It has been suggested that a package could be formed by two layers of "Surlyn A" coated with AL in between and sealed together to form a gridiron pattern or a series of squares across as well as along the length of the roll of material. Seals could easily be made across the sheet by intermittent sealing but a series of longitudinal seals would be difficult to obtain even on an intermittent basis.

Information gathered by corresponding with manufacturers of heat sealing and packaging equipment revealed that no machine is marketed that will do this job. A machine would have to be developed. However, on a small scale operation seals made in both directions by turning the sheets 90° if the length of the seal did not exceed the length of the sealing bar. Sealing bars 50" long are available.

Vacuum Formed Packages - Tray shaped package supports were made by vacuum forming. Many of the plastics ordinarily vacuum formed would have good physical properties but would not be good for the stability of the AL film. Several experiments in vacuum forming were performed to show how packages formed from "Forticel" (cellulose propionate) could be made

with an interior surface of "Surlyn A". "Forticel" (Celanese-406) 30 mils thick formed into a 12" x 12" tray had the best bottom flatness. Trays from 10 mil "Forticel" 5-1/2" x 5-1/2" had good bottom flatness and could be molded as individuals or clusters. When "Surlyn A" and "Forticel" (10 mil) were formed simultaneously over a male form the plastic sheets formed together well but did not conform to the mold as well as would be desirable. Small flat air pockets developed next to the male form. When "Surlyn A" was vacuum formed first and a sheet of "Forticel" vacuum formed over the "Surlyn", a satisfactory two layer molding was made. The "Forticel" added considerable stiffness to the tray shaped package.

A "Surlyn A" surface may be added to the bottom of a "Forticel" tray by the use of a permanently tacky film such as "Daratak" 74-L or an uncatalyzed resin such as "Epon" 828 to make the "Surlyn A" sandwich adhere. In this manner better contact is maintained between the "Surlyn" and the "Forticel". The product made by double molding above was not a fusion of the "Forticel" and the "Surlyn A" to form a laminate. Handling might cause a separation of the two layers.

"Surlyn A" (15 mil) vacuum formed to form trays 3-1/2" x 4-1/2" with increased stiffness.

3.4 CONCLUSIONS

"Surlyn A" was found the most suitable film for use in packaging AL material. It exhibited the least tendency to depress AL color appearance temperature and could be made into packages by heat sealing the AL coated film or by use as a liner in vacuum formed "Forticel" trays. Thin gloss mat was found a suitable filler sheet between the outer film sheets to prevent sag of AL in vertically hung packages and to insure uniform AL film thickness.

AL film thickness up to 10 mils provided better color observation than thicker films. A package design which permitted slight air pressure over the AL coated film provided intimate contact of the film even on relatively rough surfaces.

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SECTION IV

PROGRAM CONCLUSIONS

Feasibility has been demonstrated of utilizing anisotropic liquid films to detect soil temperature differences above a tunnel in which presence of a human being was simulated by a heat source. Practical detection of a hidden person by the technique however is doubtful because temperature difference arising from natural causes produce spurious color effects.

A packaging material and system was developed that is suitable for thermal mapping of relatively irregular and rough surfaces that are horizontal or vertical.

REFERENCES

- 1. Technical Documentary Report, ATL-TR-65-62, "Thermal Detection of Underground Tunnels", prepared under Contract Number AF 08(635)-4423 by Westinghouse Electric Corporation, Pittsburgh, Pennsylvania, September 1965.
- 2. F. A. Brooks, C. E. Barber, R. A. Kepner and C. Lorentzen, Jr., "Temperature--Its Measurements and Control in Science and Industry," Reinhold Publishing Company, New York, page 629, 1941.

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13. ABSTRACT

This work is a continuation of that reported in a ATL-TR-65-62, "Thermal Detection of Underground Tunnels," September 1965, and describes an evaluation of the feasibility of utilizing anisotropic liquid (Ab) films to detect a tunnel one foot below ground and containing a heat source which simulated the thermal effects originating from a hidden human being. Soil temperature differences above the heated tunnel were most consistent in the early morning hours just before sunrise rangeing from 0.5°F in August to 3.0°F in November. The color responses of AL panels placed in windshield crossing the tunnel plot were generally in the proper direction, however, serious difficulties with spurious color effects produced by temperature differences arising from natural causes. Mottled and variegated color patterns unrelated to tunnel location always appeared when the AL panel was placed in direct contact with the soil. At the present time it seems doubtful that the naturally occurring variations in soil surface temperatures can be eliminated to the extent necessary for practical detection of a human being hidden in a tunnel. Concurrent with this study, a series of plastic films were evaluated for potential use in packaging the AL into a unit suitable for thermal mapping applications. plastic films generally contributed to a lowering the AL color appearance temperature with Surlyn A. an Ionomer film, exhibiting the least tendency.

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